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FS(1343)BW

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: JEAN-YVES CHENARD ET AL : Group 143

U.S. Serial No. 254,313 :

Filed: April 15, 1981 :

For: IMPROVEMENT IN THE
STABILIZATION OF VINYL
HALIDE POLYMERS

AFFIDAVIT OF MICHEL FOURE
UNDER RULE 37 CFR 1.131

STATE OF NEW JERSEY)
)ss:
COUNTY OF MIDDLESEX)

MICHEL FOURE, being of legal age and under oath duly sworn, says and deposes:

That he is a chemist and has been working in the field of stabilizers for halogen containing polymers for five (5) years and has read and understands the above-identified patent application;

That prior to December 12, 1980, the following experiments were conducted in the United States on behalf of the Applicants:

EXPERIMENT

Four stabilizer compositions were prepared, each containing a mono-organo derivative of tetravalent tin where the remaining valences of the tin atom are satisfied by bonds to halogen or to halogen and sulfur. These compositions also contained a mercaptoloweralkanol ester of a carboxylic acid. Each of the four stabilizer compositions was evaluated as a stabilizer for vinyl halide polymers; a commercially available stabilizer was used as a control.

Stabilizer A

	<u>Parts by Weight</u>
Monobutyltin(isooctylmercaptoacetate)sulfide	1.82
$C_4H_9SnCl_3$	0.30
Mercaptoethyl oleate	4.93
Antioxidant ⁽¹⁾	0.25
Dioctyl phthalate	0.88

Stabilizer B

	<u>Parts by Weight</u>
$C_4H_9Sn(S)Cl$	0.93
$C_4H_9SnCl_3$	0.30
Mercaptoethyl oleate	4.93
Antioxidant ⁽¹⁾	0.12
Mineral oil ("Sunpar Oil" 150) ⁽²⁾	2.78
Dioctyl phthalate	1.77

Stabilizer C

	<u>Parts by Weight</u>
Monobutyltin(isooctylmercaptoacetate)sulfide	2.28
$C_4H_9SnCl_3$	0.31
Mercaptoethyl oleate	3.71
Antioxidant ⁽¹⁾	0.12
Mineral oil ("Sunpar Oil" 150) ⁽²⁾	2.28
Mineral oil ("Escoflex" 998) ⁽³⁾	1.30

Stabilizer D

	<u>Parts by Weight</u>
C ₄ H ₉ Sn(S)Cl	1.17
C ₄ H ₉ SnCl ₃	0.31
Mercaptoethyl oleate	3.71
Antioxidant ⁽¹⁾	0.12
Mineral oil ("Sunpar Oil" 150) ⁽²⁾	2.28
"Escoflex" 998 ⁽³⁾	2.41

(1) Available under trademark CAO-3 from Scherex Corp.

(2) Available from Sun Oil Company.

(3) Available from East Coast Chemicals.

The four stabilizer compositions described above, together with a commercially available stabilizer sold by M&T Chemicals Inc. under the trademark "THERMOLITE 310" were evaluated in a polyvinyl chloride formulation having the following composition:

	<u>Parts by Weight</u>
Vinyl chloride homopolymer ("Tenneco" 225PG) ⁽⁴⁾	100.00
Calcium carbonate ("Hydrocarb 30T") ⁽⁵⁾	3.0
Titanium dioxide	1.0
Calcium stearate	0.6
Paraffin wax ("XL 165") ⁽⁶⁾	1.0
Stabilizer	0.4

(4) Available from Tenneco Chemicals.

(5) Available from Omya Inc.

(6) Available from American Hoechst.

The formulations were evaluated using the procedure described in Example XIV of the above-identified application by fabricating sheets using a roll mill heated to a

temperature of 350°F. The sheets were removed from the mill after five minutes and then compression molded for ten (10) minutes at 350°F. Readings of the Whiteness Index (W.I.) and Yellowness Index (Y.I.) were directly obtained from a Macbeth MC 1500 colormeter.

The results of the experiment are summarized below:

<u>Stabilizer</u>	<u>W.I.</u>	<u>Y.I.</u>
A	53.9	9.7
B	57.3	8.6
C	53.0	10.0
D	55.7	9.7
T-310 (control)	36.7	15.8

The experimental results indicate that each of the four stabilizer compositions is more effective to stabilize vinyl halide polymers with respect to heat than a commercially available stabilizer; each of the four stabilizer compositions results in test specimens displaying both increased Whiteness Index and decreased Yellowness Index, compared to a commercial stabilizer.

The experiments described above were performed in the United States prior to December 12, 1980, were performed by affiant or under his direction on behalf of the Applicants, and were contemporaneously recorded by affiant in his own hand in laboratory notebook 4384 page 36, a true copy of which (dates and certain extraneous matter having been deleted) is annexed hereto as Exhibit "A".

12-19-1983

Date

Michel Foure

Sworn to and subscribed before me on the date aforesaid by Michel Foure.

Barbara Wheaton
Notary Public

BARBARA WHEATON
NOTARY PUBLIC OF NEW JERSEY
My Commission Expires March 28, 1987

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"Bu-Sn^S-Cl" will be evaluated against CN 8900 ("Bu-Sn^S-IOMA") in "B+" and "C+" formulations using the same stoichiometry for tin.

Following stabilizers are prepared:

	39-A	39-B	39-C	39-D	
CN 8900	1.82	—	2.28	—	(BWRHL-8K)
Bu-Sn(S)Cl	—	0.93	—	1.17	(3763-49)
Bu-SnCl ₃	0.30	0.30	0.31	0.31	
MEOE	4.93	4.93	3.71	3.71	(4342-04)
Caronation M.O.	1.82	—	—	—	
CAO-3	0.25	0.12	0.12	0.12	
Sunpar Oil 150	—	2.28	2.28	2.28	
Escriflex 998	—	1.50	1.30	2.41	
DOT	0.88	1.77	—	—	

* Results of press test (10min, 350°F)

Test # (4384)	WI	YI	Stabilizers	
39-1	53.9	9.7	0.4	36-A
39-2	57.3	8.6	0.4	36-B
39-3	53.0	10.0	0.4	36-C
39-4	55.7	9.2	0.4	36-D
39-5	36.7	15.8	0.4	T310

Bu-Sn^S-Cl seems to improve the WI.

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Understood by me. J.M. Grestie	Date	Invented by Michael Fournier	Date
		Recorded by	